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Bulk Properties of Powders

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THE P/M INDUSTRY has grown considerably in the past decade. As a result of this growth, more critical components in the automotive, aircraft, tooling, and industrial equipment industries are being considered for manufacture using this technology. This is placing increasingly stringent quality requirements on the final P/M part. Variations in part density, mechanical properties including strength, wear, and fatigue life, as well as in aesthetic appearance and dimensional accuracy are no longer tolerated. As a result, metal powder producers and P/M part manufacturers must continually improve their capabilities to ensure the delivery of a consistent, uniform product. Research has demonstrated that these part qualities are significantly affected by changes (variations) in the particle size distribution, particle shape, and consequently the uniformity of powder blends (a combination of one or more particle sizes of a single powder) and mixes (a combination of one or more types of powders) (Ref 1, 2).

Powder Flow

This article reviews the general factors of powder flow, and the following properties are discussed, along with examples of their applications in equipment selection:

- Cohesive strength
- Frictional properties
- Bulk density
- Permeability and flow rate
- Sliding at impact points
- Segregation tendency
- Angle of repose

The flow of metal powders in bins, hoppers, feeders, chutes, and conveyors is not always reliable or uniform. This often results in the press having to operate at lower cycle times, wasted product due to composition or apparent density variations, and operational nightmares. The powder may form a stable arch or rathole; particle segregation may occur, resulting in unacceptable variations in the bulk density of the powder supplied to the feed shoe, or the powder may flood uncontrollably.

Bulk Properties. One of the main reasons that powder flow problems are so prevalent is lack of knowledge about the bulk properties of various powders. For many engineers, the name of a powder, such as atomized aluminum, is thought to connote some useful information about its handling characteristics. While this may be true in a general sense, it is not a reliable tool. Unfortunately, major differences in flowability often occur between different grades and types of powders with the same name.

For those who go beyond the generic name of a powder, one or more of the following four attributes are often relied on in trying to predict the behavior of metal powders and other bulk solids. However, these attributes rarely provide engineers with direct assistance during the design or specification of a bin, hopper, feeder, chute, or conveyor.

Angle of Repose. Determining the angle of repose is relatively easy: simply form a pile of material and measure its slope. Knowing what to do with the data is the difficult part.

For most materials, the angle of repose varies significantly, depending on how the pile was formed. Furthermore, the mechanics of pile formation bear little resemblance to the formation of an arch or rathole in a bin or hopper, uniformity of die fill, powder homogeneity, or to the other key parameters needed when designing a material handling system. In general, the angle of repose of a material is not an accurate measure of its flowability.

Flow Rate. The Hall and Carney flowmeters (described later in this article) are widely used in the P/M industry to characterize powder flowability. However, there are two major flaws with this approach:

- If a powder will not flow through the funnel, no information on its flowability can be determined.
- Even if a powder does flow well, the value obtained (s/50 g) cannot be extrapolated to predict limiting press speed, limiting flow rate through a feed hopper, or other rate-limiting phenomena.

The attempt to combine measurements of two material flow properties (minimum orifice size and flow rate) result in a method that does not measure either one very well.

Apparent Density or Tap Density. Neither of these parameters, nor their ratio (the Hausner Ratio), is a direct indicator of powder flowability. They do not, for example, assist in sizing hopper outlets or calculating appropriate hopper angles.

Free-Flowing versus Nonfree-Flowing. Whether or not a metal powder is considered free-flowing depends to a large extent on the size and shape of the die cavity into which it is expected to flow. For example, a powder that flows through a Hall flowmeter might be considered free-flowing; however, that same powder may have difficulty completely filling a die cavity for a thin-wall part. Thus, these terms are relative and not absolute indicators of powder flowability.

Flow Pattern Considerations. There are two flow patterns that can develop in a bin: funnel flow and mass flow. Both patterns are shown in Fig. 1.

In funnel flow, an active flow channel forms above the outlet with nonflowing material at the periphery. As the level of material in the hopper decreases, layers of the nonflowing material may or may not slide into the flowing channel, which can result in the formation of stable ratholes. In addition, funnel flow can cause product caking, provide a first-in last-out flow sequence, and increase the extent to which sifting segregation impacts the discharging material.

In mass flow, all of the material is in motion whenever any is withdrawn from the hopper. Material from the center as well as the periphery moves toward the outlet. Mass flow hoppers provide a first-in first-out flow sequence, eliminate stagnant material, reduce sifting segregation, provide a steady discharge with a consistent bulk density and a flow that is uniform and well controlled. Requirements for achieving mass flow include sizing the outlet large enough to prevent arching and ensuring the hopper walls are sufficiently smooth and steep enough to promote flow at the walls.

Useful Bulk Flow Parameters. Armed with information about the bulk properties of the powder, engineers can optimize the selection of storage and handling equipment. These same

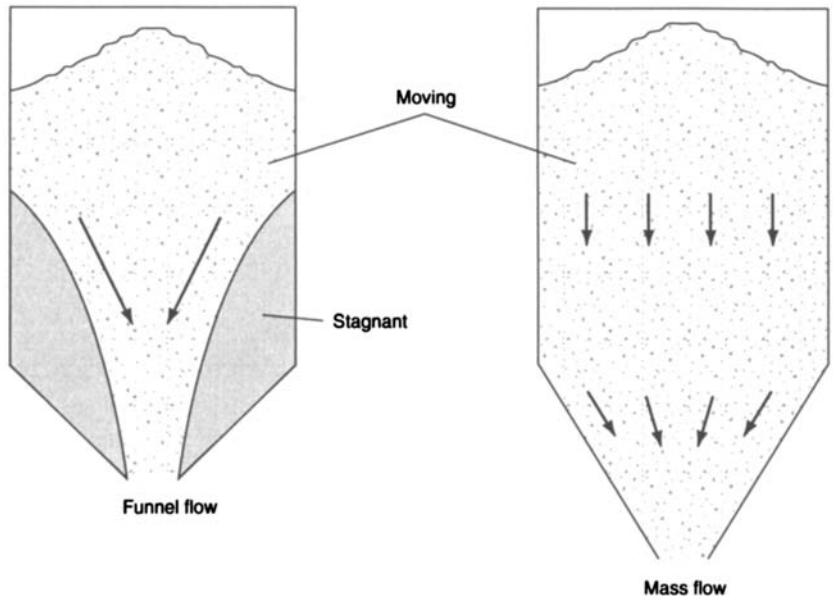


Fig. 1 Two flow patterns that can occur in a bin: funnel flow and mass flow

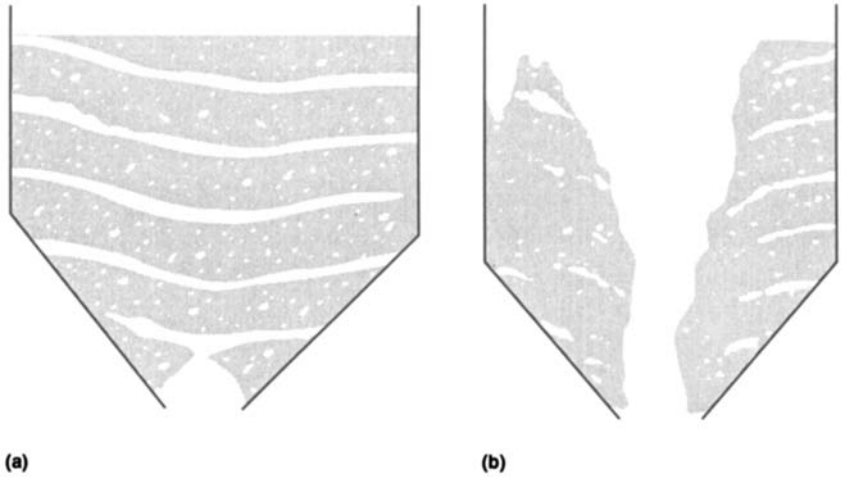


Fig. 2 Examples of no-flow situations where the darkened areas represent material within the bin. (a) Cohesive arch at the outlet of a bin. (b) Stable rathole formed within bin

properties can be used to retrofit existing processes to correct flow problems.

Discussed below are several bulk solids handling properties that are relevant to predicting flow behavior. The direct application of these parameters has been proven over the last 30 years in numerous installations handling the full spectrum of powders used in the P/M industry, including metal powders, fine chemical additives, polymers and waxes, and graphites/carbons (Ref 3, 4).

Cohesive Strength

Many metal powders and other bulk solids, when poured from a box, flow like a liquid. Under these conditions, such a material has no cohesive strength. However, when squeezed in the palm of one's hand, the material may gain enough strength to retain its shape once the hand is opened.

A similar range of conditions occurs inside bins, hoppers, and containers. Consolidation pressures range from zero at the surface to relatively large values at increasing depth within the container. If a powder gains cohesive strength because of the pressures applied to it, an arch or rathole can form (Fig. 2).

An arch (also called a bridge or dome) is a stable obstruction that forms over the point of narrowest cross section of the storage vessel (usually the discharge outlet). The arch supports the rest of the bin contents, preventing discharge.

A rathole is a stable pipe or vertical cavity that empties out over the outlet. Material is left stranded in stagnant zones that usually remain in place until an external force is applied to dislodge them.

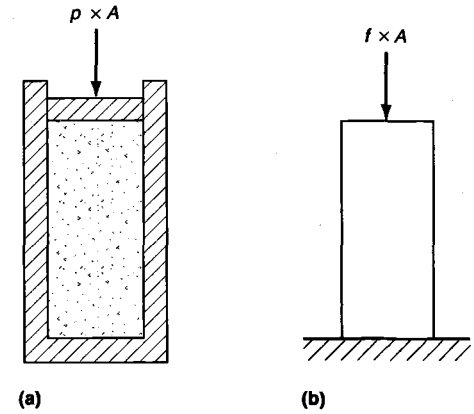


Fig. 3 An idealized flow function test. (a) Consolidation. (b) Failure

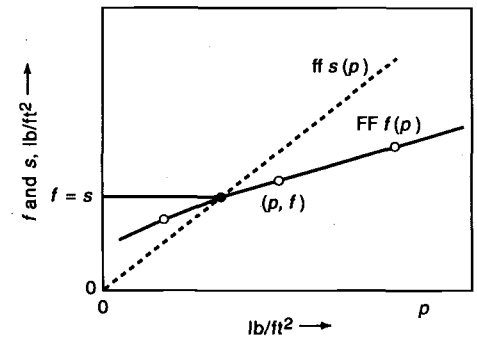


Fig. 4 Solids flow function (FF) and hopper flow factor (ff). See text for details.

Cohesive strength can be measured as a function of the applied consolidation pressure. This relation, which is of primary importance in the analysis of flow, can be described as follows: suppose that a quantity of a metal powder has been placed in a cylindrical mold of cross-sectional area A, with frictionless walls (Fig. 3), and consolidated under a force ($p \times A$) applied to the piston. Now suppose that, without disturbing it, the consolidated cylinder of powder is removed from the mold and placed on a table, and a compressive force is applied to it (Fig. 3). The force is increased from zero until the cylinder collapses at some value of the force ($f \times A$). The experiment is repeated for several values of p. For each value of p, a corresponding value of f is obtained. Points (p, f) are plotted in Fig. 4, and a smooth line is drawn through them. This relation is called the flow-function (FF) of the solid.

This compression test serves well as an illustration of the concept, but is not practical for a number of reasons; for instance, a mold cannot be made frictionless, and it is difficult to obtain uniform consolidation of a powder in a relatively tall cylinder. In addition, most metal powders have such low cohesive strength that the compacted cylinder of powder would fall apart when it was removed from the cylindrical mold.

A more accurate and controlled procedure is described in ASTM D 6128 (Ref 5). In a laboratory, a sample of the powder is placed in a Jenike

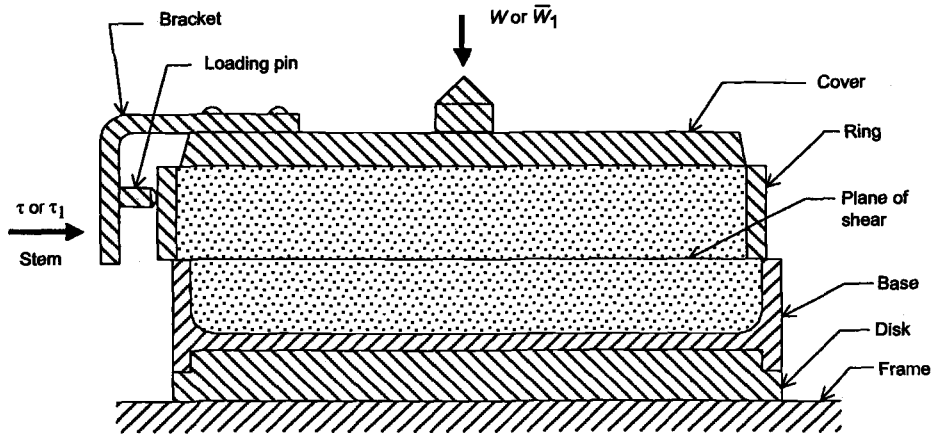


Fig. 5 Jenike shear cell in initial offset shearing position

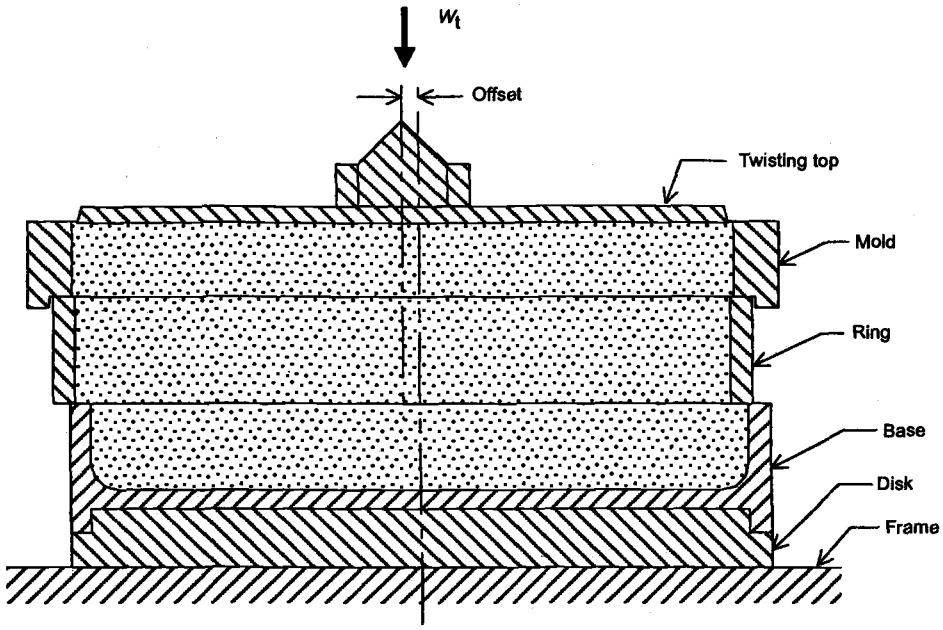


Fig. 6 Jenike shear cell with mold ring and consolidation lid set up for pre-consolidation

shear cell (Fig. 5), and both compressive and shear loads are applied to simulate flow conditions in a container.

The shear cell, Fig. 5, is composed of a base located on the frame of the machine, a ring resting on top of the base, and a cover. The bottom of the cover and the inside of the base are roughened to increase friction with the tested powder. The base and the ring are filled with the powder to be tested, and a vertical load is applied to the cover. A horizontal shearing force is applied by means of a stem, which acts on a bracket attached to the cover. This shearing force, which acts in the plane of contact between the ring and the base, is partly transferred from the bracket to the ring through a loading pin. This ensures a sufficiently uniform distribution of the shearing force across the cell. The standard shear cell is 95 mm (3.75 in.) inside diameter, but a 65 mm (2.5 in.) diameter is also often used.

A shear tester is equipped with a shear cell, a gravity vertical loading system, and an electronic shearing force applicator. The applicator in the Jenike shear tester has a shearing rate of 2.54 mm/min (0.10 in./min). The shearing force necessary to maintain the strain rate is continuously recorded on a strip-chart recorder. This arrangement produces a permanent record of the stress-strain relations for each test.

Following the simplified example (described above) of testing using a cylindrical mold, shear testing is a two-step process involving consolidation (also called preshear) and shear.

Consolidation is carried out in two stages. The purpose of the first stage, called preconsolidation, is to prepare a uniform specimen. With the cover off the test cell, a packing mold is placed on top of the ring, and both the mold and the ring are placed in an offset position on the base, as shown in Fig. 6. A sample of the tested powder is then placed in the cell. One layer after another is slightly packed with the fingers, up to the top of the mold. The excess material is scraped off level with the top of the mold, and a twisting top is placed over the powder. A vertical force is applied to the top by means of a weight hanger. This force causes a vertical pressure σ_t in the material. By means of a special wrench, a number of oscillating twists is applied to the cover. This preconsolidates the powder and ensures a uniform specimen.

Consolidation is completed in the second stage by causing the specimen to flow under given stresses until a steady state is reached, or closely approached. This is attained in the following way: The twisting load is taken off, the twisting top and the mold are removed, the excess material is scraped off level with the top of the ring, and the test cover is placed on the material. A smaller load is now placed on the cover, and the stem of the shearing device is advanced against the bracket (Fig. 5). This smaller load compacts the sample to a preshear normal stress of σ_p.

As shearing proceeds, a condition is reached when a layer of the powder across the whole specimen is caused to flow plastically: the recorded shearing stress reaches a steady value τ_p. Consolidation determines point (σ_p, τ_p), (Fig. 7).

Shear. When consolidation is completed, the stem of the shearing force device is retracted. The preshear normal stress σ_p is replaced by a smaller normal stress σ_s to locate a useful point (σ_s, τ_s) of the yield locus (Fig. 7). The sample is now sheared until a failure plane has developed. This fact is indicated on the recorder by the stress τ_s passing a maximum value. After shearing, the plane of failure of the specimen is checked. It should roughly coincide with the plane of shear of the cell. If the planes deviate, it means that the measured point (σ_s, τ_s) does not lie on the yield locus, and the test is repeated.

The determination of one yield locus requires the measurement of three to five points of the locus $(\sigma_s, \tau_s)_1$, $(\sigma_s, \tau_s)_2$, $(\sigma_s, \tau_s)_3$ and so forth (Fig. 7). For each point, the specimen is first consolidated and then sheared. The value of shear normal stress σ_s typically ranges between 25 and 80% of the preshear normal stress σ_p. It is necessary to obtain the values of these points for the same steady consolidating shear each time. This is accomplished by running a sufficient number of tests to permit interpolation to a suitably selected value of τ_p.

The yield locus is now drawn and extrapolated toward the higher value of σ_p, and a Mohr semicircle is drawn through point (σ_p, τ_p) tangentially to the yield locus. The point of tangency (E) locates the terminus of the yield locus. The point of intersection of the semicircle with the σ-axis determines the value of the major consolidation stress, σ_1. The Mohr semicircle for the unconfined yield strength f_c is now drawn. The value of f_c is determined by the point of intersection of the circle with the σ axis, as shown in the Fig. 7.

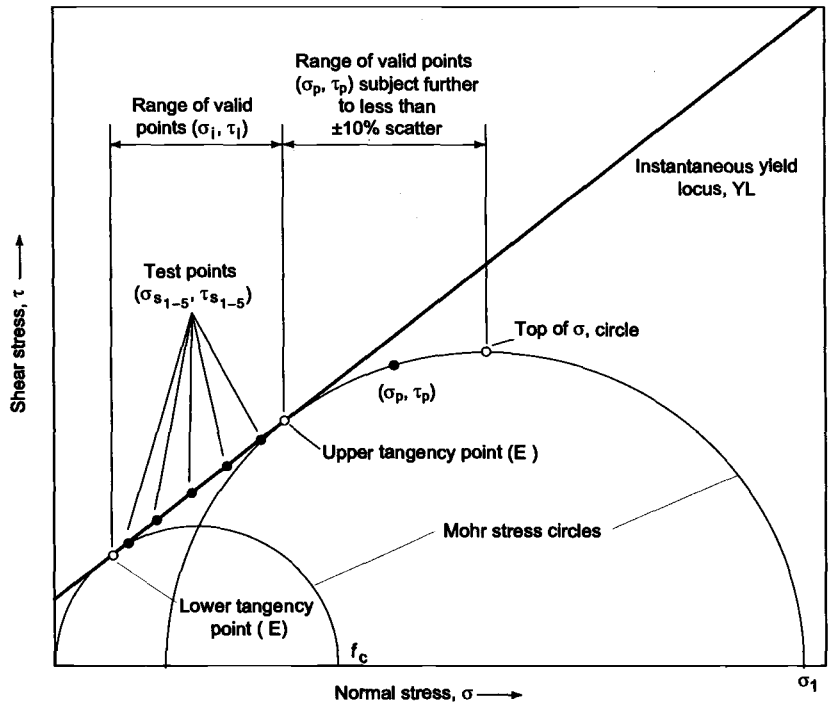


Fig. 7 Yield locus (YL) showing valid shear testing points

A plot of f_c versus σ_1 is the flow function of the powder under the conditions of particle size, moisture, and temperature tested.

To simulate time of storage at rest, the shear tester is used in conjunction with a six-cell consolidating bench (Fig. 8). The tester shown in Fig. 9 is used for temperature sensitive solids. Here, the consolidating bench is enclosed in a heated chamber that permits the control and recording of the temperature of the tested powder.

The flow function of a powder is used for a variety of engineering analyses, for example, to calculate minimum outlet dimensions required to prevent cohesive arches and ratholes from forming. Details can be found in Ref 3.

The cohesiveness of a bulk solid is a function of the following parameters:

- *Moisture:* Typically, cohesiveness rises as moisture content increases, although not in direct proportion. Hygroscopic materials can experience significant increases in moisture when exposed to humid air.
- *Particle size and shape:* There is no direct correlation between particle size, shape, and cohesiveness. Even so, in most cases, as a powder becomes finer, it also becomes more cohesive and difficult to handle. Angular or fibrous particles are often more cohesive than those that are rounded.
- *Temperature:* The temperature of a powder can affect its cohesiveness. For example, many thermoplastic blends (such as for PIM) become more difficult to handle as their temperatures rise. Some materials have more strength at constant temperatures, while others gain cohesive strength as their temperature changes during heating or cooling.
- *Time of storage at rest:* When a material resides in a bin or hopper for a period without moving, it can become more cohesive and difficult to handle. Such cohesion may be caused by settling and compaction, crystallization, chemical reactions, and adhesive bonding.
- *Chemical additives:* In some cases, adding a small amount of a chemical additive such as calcium, lithium, or zinc stearates can cause a cohesive powder to flow more easily.

Frictional Properties

Both internal and external friction values are important when characterizing the flow properties of a metal powder. Internal friction is caused by solid particles flowing against each other and is expressed by the angle of internal friction and the effective angle of internal friction. Both can be determined during the course of measuring cohesive strength with a Jenike shear cell (Fig. 5), as described in Ref 5.

External friction is expressed as the wall friction angle or coefficient of sliding friction. The lower the coefficient of sliding friction, the less steep the hopper walls need to be for powder to flow along them (mass flow). Also, the easier a feed shoe indexes to and from a die, the more uniform the flow of powder into the die cavity.

The coefficient of sliding friction can be measured by sliding a sample of powder across a stationary wall surface using a shear tester. The arrangement of the cell is shown in Fig. 10. In this case, a coupon of the wall material is placed on a filler so that the top surface of the coupon is the horizontal plane of the force measuring stem. The ring and packing mold are placed over the wall material coupon and filled with the powder.

After scraping off the excess material level with the top of the mold, a twisting top is placed over the powder. A vertical force is applied to the top by means of a weight hanger. This force causes a vertical pressure σ_t in the material. By means of a special wrench, a number of oscillating twists is now applied to the cover. This pre-

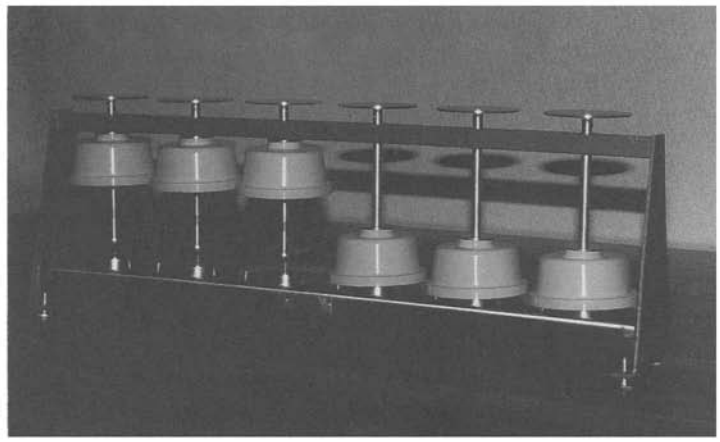


Fig. 8 Consolidation bench

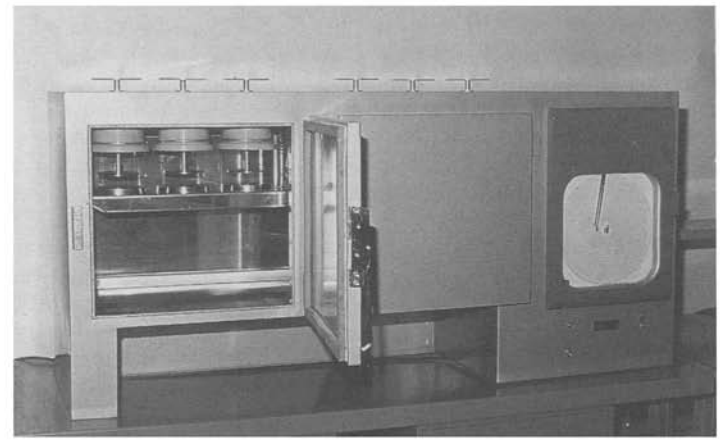


Fig. 9 Consolidation bench in heated chamber

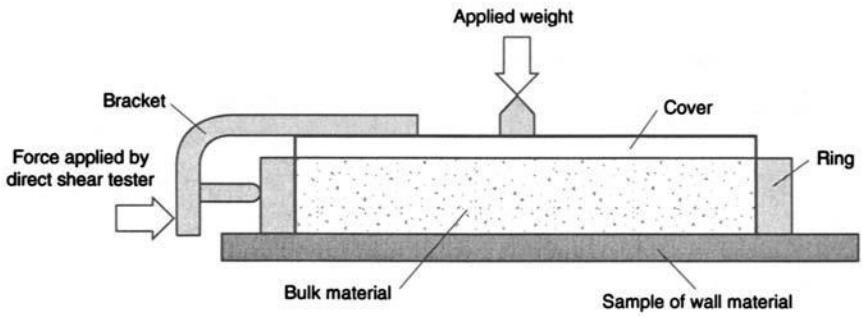


Fig. 10 Shear cell used in measuring wall friction properties. Test setup design allows shear stress (see horizontal arrow) to be measured as a result of applied weights (see vertical downward arrow).

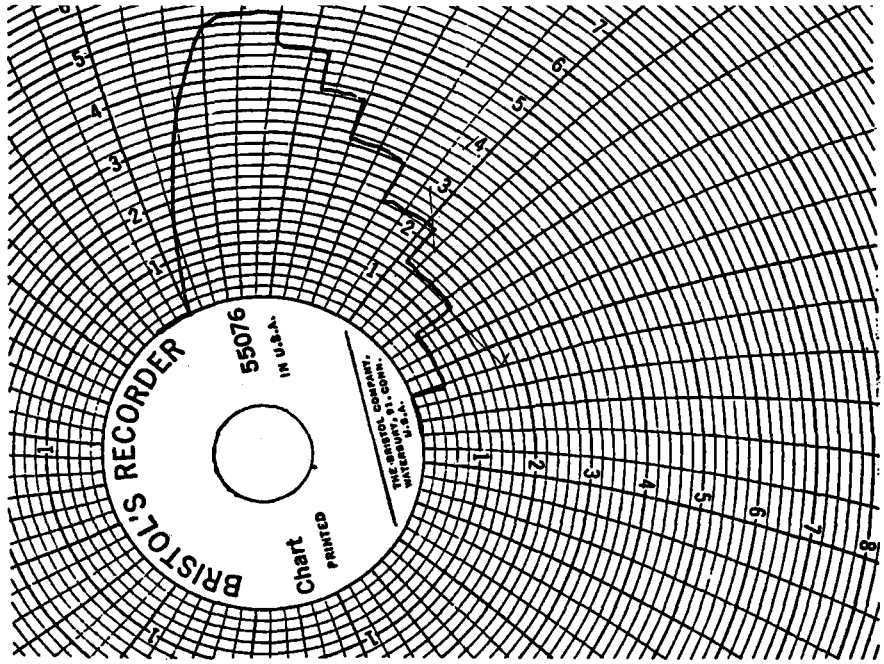


Fig. 11 Typical recorder chart in measurement of ϕ' (wall friction angle)

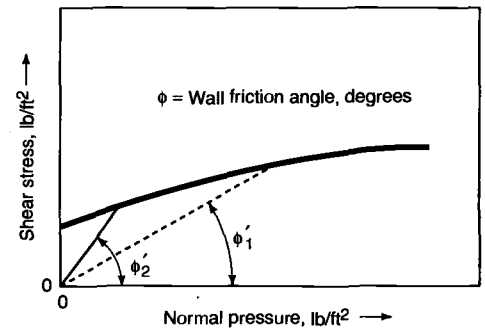


Fig. 12 Typical results of the test setup shown in Fig. 10 to help engineers determine wall friction angle (ϕ')

consolidates the powder and ensures a uniform specimen.

The twisting load is taken off, the twisting top and the mold are removed, the excess material is scraped off level with the top of the ring, and the test cover is placed on the material. A smaller load is now placed on the cover, and the stem of the shearing device is advanced against the bracket (Fig. 10). All the tests necessary to determine the coefficient of sliding friction are now run without replacing the powder.

Before the start of a test, the ring is twisted and manually lifted slightly off the wall material coupon to prevent it from dragging on the wall coupon. Several (say, six) one or two pound weights are placed directly on top of the cover of the shear cell to give the largest required normal stress σ_w. The stem is advanced. When the shear stress τ_w has leveled off, one weight is removed, after a while τ_w again levels off, another weight is removed and so on, until all the weights have been removed. The cover, ring, and the enclosed powder are then weighed. Their weight plus the superimposed weights determine the normal stresses σ_w.

A typical recorder chart is shown in Fig. 11. The points (σ_w, τ_w) are plotted in Fig. 12. A smooth line drawn through these points is the wall yield locus, WYL. Typically, the WYL is convex upward.

The coefficient of sliding friction is the ratio of the shear force required to cause sliding to the load applied perpendicular to the wall material coupon. The arc tangent of this value is the wall friction angle (Ref 3, 4).

The following variables can affect the internal and external friction values of a metal powder and are similar to those affecting cohesiveness:

- *Pressure:* Typically, as consolidating pressure increases, the effective angle of internal friction decreases. Similarly, the coefficient of sliding friction often decreases as pressure acting normal to the plate increases. However, the internal angle of friction is an intrinsic characteristic of the material that may increase, decrease, or remain the same as pressure acting on the material increases.
- *Moisture content:* As moisture increases, many bulk solids become more frictional.
- *Particle size and shape:* Typically, fine materials and those with a wide range of particle sizes are somewhat more frictional than coarse materials or those with a narrow particle size distribution; so the flow of the former is often more troublesome. Shape plays a role in that angular particles tend to interlock and also dig into a wall surface, thereby creating more friction.
- *Temperature:* For many materials, higher temperatures can cause particles to become more frictional.
- *Time of storage at rest:* If allowed to adhere to a wall surface, many powders experience an increase in friction between the particles and the wall surface. Such situations require steeper bin walls for unaided flow.
- *Wall surface:* The initial condition of a surface can play a major role in how materials slide along it. Smoother surfaces are typically less frictional, although this is not always true. Also, as a carbon steel container ages, corrosion can roughen the walls, making sliding more difficult.

Friction data are used to:

- *Design a mass flow hopper:* Values of both wall friction angle and effective angle of internal friction are required to design a mass flow hopper. Using these angles along with design charts given by Jenike (Ref 3), one can determine allowable hopper angles required to promote mass flow.
- *Anticipate sliding on chutes:* A chute is used to transfer material from one point to another in a bulk handling system. By definition, the cross section of a chute is only partially full at any given time, and the discharge rate of a powder is equal to the chute filling rate.

Beyond the impact point, the acceleration of a particle on a chute is directly related to the difference between the wall friction angle of the material and the chute angle. As long as the chute is steeper than the wall friction angle, particles will continue to accelerate. Otherwise they will slow down and may eventually block the chute.

Table 1 Effect of particle size on apparent density for several metal powders

Material	Average particle diameter(a), μm	Apparent density, g/cm³
Aluminum		
Atomized	5.8	0.62
	6.8	0.75
	15.5	0.98
	17.0	1.04
	18.0	1.09
	60% above 44 (+325 mesh)	1.22
	75% above 44 (+325 mesh)	1.25
Copper		
Electrolysis	90% min, −325 mesh	1.5–1.75
Hydrometallurgical	81.9%, −325 mesh	1.69
Oxide reduced	95% min, −325 mesh	2.10–2.50
Hydrometallurgical	49.1%, −325 mesh	2.42
Oxide reduced	50–65%, −325 mesh	2.65–2.85
Electrolysis	60–75%, +100 mesh	4.0–5.0
Atomized	70% min, −325 mesh	4.9–5.1
	50–60%, −325 mesh	4.9–5.5
Nickel		
Carbonyl	3.2	0.61
Precipitation	3.5	1.81
Carbonyl	3.8	1.87
	4.1	2.10
Precipitation	4.4	2.09
	8.0	2.60
	−40+325 mesh	3.60
Tungsten		
Oxide reduced	1.20	2.16
	2.47	2.52
	3.88	3.67
	6.85	4.40
	26.00	10.20
Stainless steel		
Atomized, spherical	−325 mesh	4.3
	−270+325 mesh	4.5
	−200+270 mesh	4.4
	−150+200 mesh	4.5
	−100+150 mesh	4.5
Iron		
Reduced	6	0.97
Carbonyl	7	3.40
Reduced	51	2.19
Electrolytic	53	2.05
	63	2.56
Reduced	68	3.03
Electrolytic	78	3.32

(a) From Fisher subsieve sizer for single values and screens for size fractions

Bulk Density

Bulk density measurements of powders include apparent density and tap density measurements as described below. A key factor is compressibility.

Apparent Density

Apparent density of a metal powder, or the weight of a unit volume of loose powder expressed in grams per cubic centimeter, is one of the fundamental properties of a powder. This characteristic defines the actual volume occupied by a mass of loose powder, which directly affects processing parameters such as the design of compaction tooling and the magnitude of the press motions required to compact and densify loose powder.

Table 2 Effect of mixture of spherical coarse and fine stainless steel particles on apparent density

Particle size (mesh)	Particles, %					
−100+150	100	80	60	40	20	...
−325	...	20	40	60	80	100
Apparent density, g/cm³	4.5	4.9	5.2	4.8	4.6	4.3

Table 3 Apparent densities and flow rates of electrolytic iron powders of three particle size distributions

Particle size (mesh)	Particles, % Powder A	Powder B	Powder C
+100	4	3	15
−100+150	11	26	10
−150+200	18	18	30
−200+250	16	6	25
−250+325	18	16	5
−325	33	31	15
Apparent density, g/cm³	2.6–2.8	3.2–3.4	3.8–3.9
Flow rate, s/50 g	29	24	20

In most compacting operations, dies are filled by volume measure, and presses operate either to a fixed position or a fixed pressure. If the press operates to a fixed position, pressure can be maintained at a constant level only if the apparent density of the powder does not change. If, however, the press operates to a fixed pressure, consistency in apparent density is necessary to ensure compacts of equal height. Small fluctuations in apparent density can be compensated for by adjustments of pressure or stroke of the presses, but large-scale compacting requires that the apparent density of the powder be controlled within close limits.

Factors Affecting Apparent Density. Apparent density of a metal powder depends on the density of the solid material, particle size, particle size distribution, particle shape, surface area and roughness of individual particles, and particle arrangement. Apparent density is strongly affected by particle size. It generally (1) decreases with decreasing particle size, (2) decreases as the particle shape becomes less spherical and more irregular, (3) decreases with increasing surface roughness, and (4) is frequently controlled by mixing various sizes of particles.

Particle Size. Decreasing particle size generally decreases apparent density. The smaller the particles, the greater the specific surface of the powder. This phenomenon increases the friction between particles and subsequently decreases the apparent density. Powder particles that exhibit very low friction because of their rounded shape, such as gas-atomized (spherical) stainless steel powder, do not demonstrate this characteristic. The effect of decreased particle size on density is particularly significant for particle sizes of less than 20 μm. Table 1 shows the effect of particle size on apparent density for several metal powders.

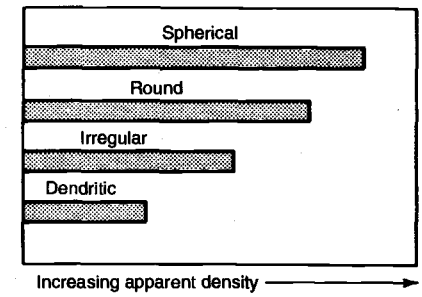


Fig. 13 Effect of particle shape on apparent density of a metal powder

Particle Shape. As particle shape becomes less spherical, apparent density decreases, due to both the increase in frictional surface area and less uniformity of powder particles during packing. Spherical powders, which are normally produced by atomizing, frequently have high apparent densities, about 50% of the density of the wrought metal. Spheres are most likely to pack without bridging or arching to create empty spaces; they tend to move easily past each other because of their smooth surfaces. At the other extreme in particle shape are flake powders, which often have apparent densities less than 10% of the wrought density. These powders are useful primarily as pigments, because their low apparent density aids in obtaining mixtures in paint.

Most powders used for compacting have irregular, somewhat equiaxed particle shapes with apparent densities that fall in the range between those of spherical and flake powders. Apparent densities of these particles range from 25 to 35% of the wrought density of the metal. Figure 13 illustrates the effect of particle shape on apparent density.

Surface Roughness. Decreasing surface area-to-volume ratios and decreasing surface roughness tends to reduce frictional forces between settling particles. This tendency thus increases apparent density by allowing the particles to move more effectively to fill the free spaces between previously settled particles.

Particle Size Distribution. An effective way to increase the apparent density of a powder is to fill the spaces between particles with smaller particles. Figure 14 shows the effects of adding differently shaped −325 mesh powder to a standard +325 mesh blend of stainless steel powder. Table 2 shows this effect for mixtures of fine and coarse spherically shaped stainless steel powders, where a mixture of about 60% coarse and 40% fine particles is optimal.

The addition of fine spherical powder effectively increases apparent density, while the opposite is true of flake powders. Distribution of a variety of particle sizes greatly affects apparent density. The relative amount of coarsest and finest particles and the percentage of particles between the two extremes determine apparent density. An example of this is shown in Table 3 for three particle size distributions.

Hall Flowmeter and Carney Funnel. The most common method for determining apparent

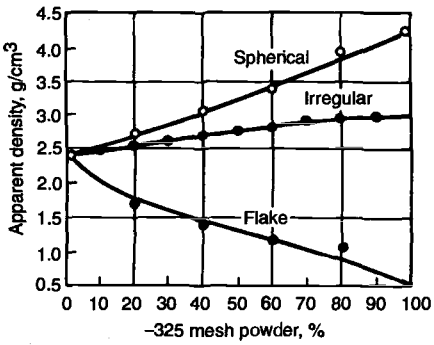


Fig. 14 Effect of three different shapes of -325 mesh powder addition to a +325 mesh distribution on apparent density of 316 stainless steel powder

density of metal powders uses the Hall flowmeter. Both ASTM B 212 and Metal Powder Industries Federation (MPIF) standard 04 describe this method.

Critical equipment dimensions are illustrated in Fig. 15(a), (c), and (d). Apparent density determinations are made by pouring powder into the funnel and allowing it to flow into the 25 cm^3 (1.5 $in.^3$) density cup. After the cup is fitted, the funnel is moved away and the excess powder is carefully leveled off using a spatula or straight edge. Care must be exercised to prevent physical densification of the powder in the cup when leveling. The apparent density in grams per cubic centimeter is then determined by weighing the powder in the cup in grams and dividing by 25 cm^3 (1.5 $in.^3$) (cup volume).

For powders that do not flow freely, a second method, described in ASTM B 417 and MPIF 28, has been devised. This is similar to the Hall flowmeter procedure, except that a different funnel, the Carney funnel, which has an orifice diameter twice that of the Hall funnel, is used (see Fig. 15a and b). This larger opening permits a greater variety of powders to flow. Powders that do not flow readily can be freed by poking a wire up and down in the hole. The wire must not enter the density cup at any time. This second method is fast and correlates well with the Hall flowmeter evaluation of free-flowing metal powders.

A wire is not used with the Hall flow-meter funnel because it may scratch the orifice and ruin the calibration of the funnel for flow tests. The Carney funnel is often used when measuring apparent density of lubricated powders because lubricant adhering to the smaller orifice of the Hall funnel temporarily affects the calibration of the Hall flowmeter.

Scott Volumeter. Another instrument frequently used for determining apparent density is the Scott volumeter, described in ASTM B 329, which was originally developed by Scott, Schaeffer, and White for the determination of the density of dry pigment for paint. As shown in Fig. 16, the device consists of:

- *A large brass funnel* with a metal screen and a smaller funnel with a straight stem for directing the powder into the baffle box

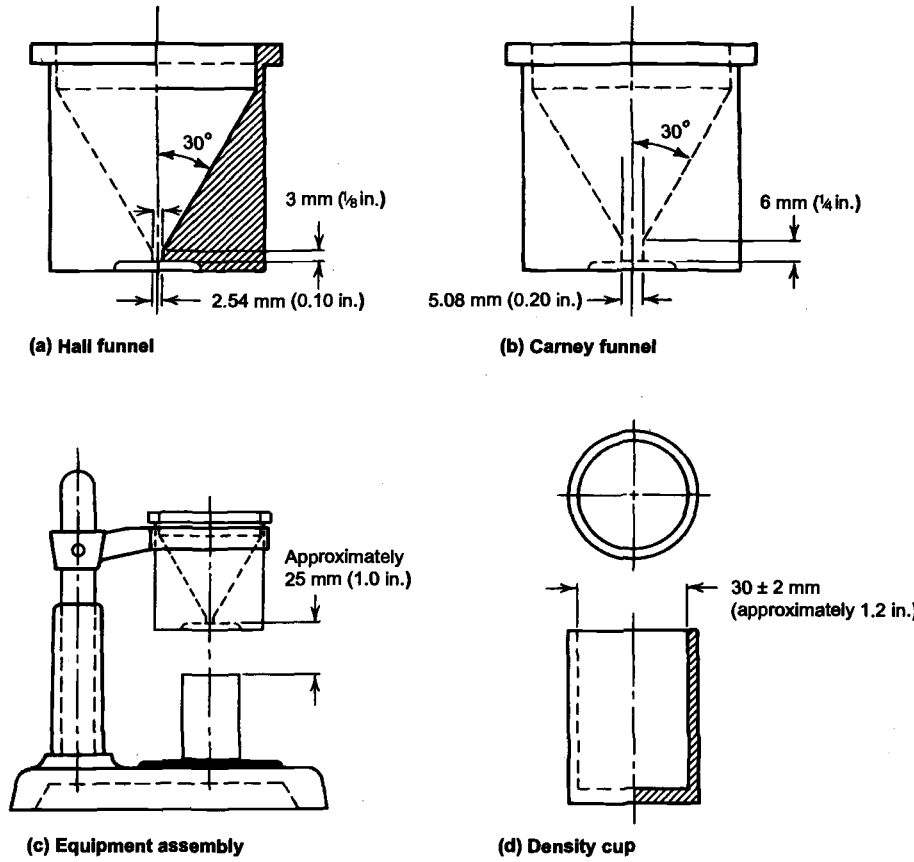


Fig. 15 Dimensions for apparent density equipment

- *A baffle box* with glass sides and two wooden sides containing a series of glass baffle plates and a funnel at the bottom to collect the powder and direct it into the density cup
- *A square density cup* with a capacity of 16.4 ± 0.032 cm^3 (1 ± 0.002 $in.^3$) or a cylindrical cup with a capacity of 25.00 ± 0.05 cm^3 (1.5 ± 0.003 $in.^3$) with an inside diameter of 30.00 ± 2.00 mm (1.2 ± 0.08 in.)
- *A stand* to support the funnels and baffle box concentric with the density cup, so that the bottom of the baffle box funnel is 19 mm (0.75 in.) above the top of the density cup when the apparatus is assembled
- *A level, vibration free base* to support the funnels and baffle box
- *An analytical balance* having a capacity of at least 100 g (3.5 oz) and a sensitivity of 0.1 g (0.0035 oz)

Operating Procedure. The test specimen is carefully poured into the funnel. Ultrafine powders may require light brushing with a nylon brush to initiate powder flow through the screen in the funnel. Powder is allowed to run into the density cup until it completely fills and overflows the periphery of the cup. The funnel and baffle box should then be rotated approximately 90° in a horizontal plane to clear the cup.

Excess material should be removed from the cup by passing a spatula blade in flat contact with the top of the cup. The spatula is moved smoothly back and forth along the top of the cup until all excess powder has been removed. When insufficient powder is left for the first reverse pass to smooth the surface completely, powder on the spatula should be gently replaced on top of the cup. The spatula must be kept level at all times to prevent packing or pulling out of the powder.

After the leveling operation, the side of the density cup should be tapped lightly to settle the powder to avoid spilling. The powder is transferred to a balance and weighed to the nearest 0.1 g (0.0035 oz). The density of the powder in the density cup is given in grams per cubic centimeter if a metric cup is used, or grams per cubic inch if a nonmetric cup is used.

Arnold Meter. Another device developed to determine the apparent density of metal powders is the Arnold meter, described in ASTM B 703 and MPIF 48, which is designed to duplicate the action of a feed shoe filling a die cavity on a P/M press. A hardened, fully demagnetized steel block with a round hole having a volume of 20 cm^3 (1.2 $in.^3$) is placed on a sheet of glazed paper. A bronze bushing filled with powder is slid across the hole. The powder collected in the hole is then removed and weighed. Apparent density is calculated by dividing the weight by the volume of the hole.

Apparent densities obtained with this procedure, which takes less than 5 min to perform, are quite close to those values measured on powder that has filled a die cavity from a filling shoe in an automatic compacting press.

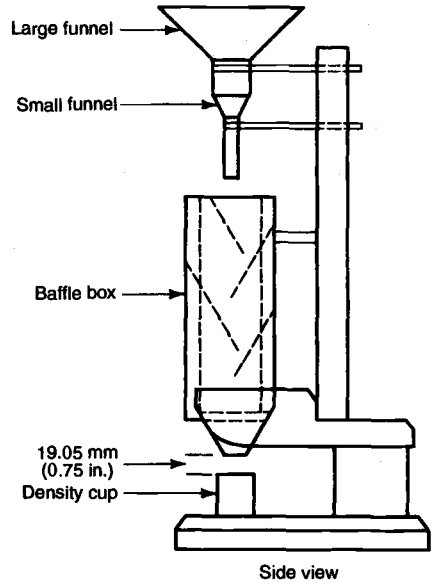


Fig. 16 Scott volumeter

Tap Density

Tap density is defined as the density of a powder when the volume receptacle is tapped or vibrated under specified conditions. Tapping or vibrating a loose powder induces movement and separation and lowers the friction between the powder particles. This short-term lowering in friction results in powder packing and in a higher calculated density of the powder mass. Tap density is always higher than the free-flow apparent density.

Tap density is a function of particle shape, particle porosity, and particle size distribution. It is commonly included as a control specification for metal powder, but is used in other industrial applications as a practical measure of the degree of powder packing that occurs in containers.

The amount of increase from apparent to tap density depends to a great extent on particle shape. Table 4 compares the density increases for three types of copper powders. Usually, the lower the apparent density, the higher the percentage increase in density on tapping.

Equipment and Test Procedures. Three pieces of equipment are needed to determine tap density:

- A balance with the capacity of weighing up to 100 g with an accuracy of 0.1 g
- A graduated glass cylinder with a capacity of 100 mL and an accuracy of 0.2 mL (or a smaller graduated cylinder may be used for high-density powders)
- A mechanical apparatus, such as the Tap-Pak volumeter, capable of tapping the graduated cylinder at a rate of 100 to 250 impacts per minute or, alternatively, a hard rubber slab approximately $100 \times 100 \times 5$ mm ($4 \times 4 \times \frac{1}{4}$ in.)

To determine tap density, a standard weight (usually 50 g) of powder is weighed to ±0.01 g.

Table 4 Effect of particle shape of copper powders

Particle size distribution is the same for apparent and tap density values

Particle shape	Apparent density, g/cm^3	Tap density, g/cm^3	Increase, %
Spherical	4.5	5.3	18
Irregular	2.3	3.14	35
Flake	0.4	0.7	75

Source: Ref 6

The powder is poured into a clean, dry graduated cylinder, taking care that a level surface of powder is obtained. For refractory metal powders that have high apparent densities (above 4 g/cm^3), it is preferable to use a reduced-volume graduated cylinder (25 mL) to improve the accuracy of the results.

The powder is settled in the cylinder by mechanical or hand tapping. If mechanical tapping (Fig. 17) is used, the filled cylinder is placed in the mechanical apparatus, which is operated until no further decrease in the volume of the powder is observed. If hand tapping is used, the base of the filled cylinder is tapped squarely on a hard rubber slab until no further decrease in volume is observed. Care must be exercised to avoid loosening the surface layers of the sample during this procedure.

The volume of the fully densified powder sample in the graduated cylinder is read and used in the following calculation of tap density:

$$\text{Tap density } \rho_t = \frac{m}{v}$$

where m is the mass of powder in grams and v is the volume of tapped powder in cubic centimeters. Results should be reported to the nearest 0.1 g/cm^3.

Representative tap densities of several metal powders and compounds are listed in Table 5, with their respective Fisher subsieve sizes. Mechanical tapping is performed in accordance with ASTM B 527, MPIF 46, or International Standards Organization (ISO) standard 3953. Manual tapping is performed in accordance with either the MPIF or ISO standard. Interlaboratory reproducibility of tap density values can be expected to fall within a standard deviation of about 3.5%.

Compressibility

In most cases, the bulk density of a material varies continuously as a function of the consolidating pressure acting on it. Therefore, it is not sufficient to describe a material simply in terms of its apparent density or tap density. Instead, this density-to-pressure relationship can be measured (Ref 7), and the results are often expressed as a straight line on a log-log plot (Fig. 18). In the bulk solids literature, this relationship is often called compressibility, although this term has another definition in the P/M industry.

The following variables can affect the bulk density of a material:

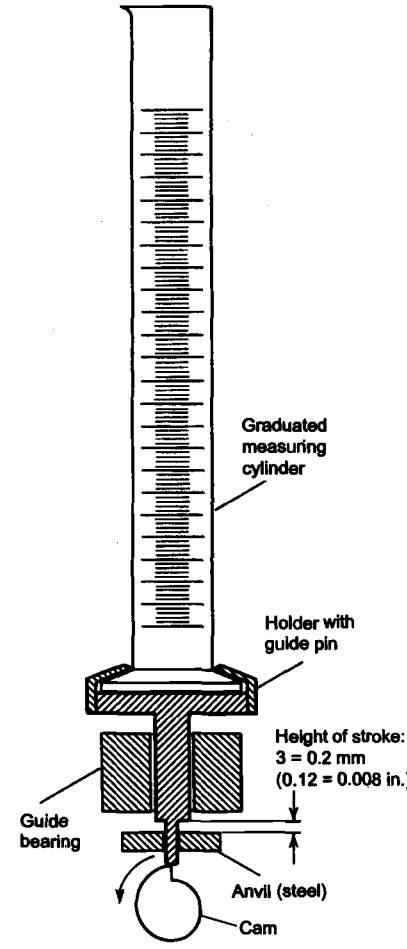


Fig. 17 Diagram of tapping apparatus

- *Moisture:* Higher moisture content usually makes a material more compressible.
- *Particle size and shape:* Often, the finer the bulk solid, the more dense and compressible it is. The shape of the particles can affect how they fit together, as well as their tendency to shear while being compacted.
- *Temperature:* Some materials become more compressible as their temperature increases.
- *Particle elasticity:* Elastic materials tend to deform significantly when they are compressed.

Some of the uses of compressibility data are:

- *Wall friction angle:* Bulk density values at various points in a hopper are used to calculate the pressures acting perpendicular to the hopper wall. After running a wall friction test, the wall friction angle is determined for a variety of pressures and used to calculate limiting angles in a mass flow hopper.
- *Feeder design:* To calculate the loads that act on a feeder or gate, one must know the bulk density of the material at the hopper outlet. Knowing this density also helps in sizing a volumetric feeder and choosing its speed.

Table 5 Typical tap densities of metal and metal carbide powders

Powder	Fisher subsieve size, μm	Tap density, g/cm³
Aluminum	5.05	1.30
Chromium	3.20	3.10
Chromium carbide (Cr_3C_2)	3.70	3.50
Cobalt	1.50	1.60
Hafnium carbide (HfC)	3.50	5.95
Iron	5.40	3.55
Manganese	3.40	3.05
Molybdenum	4.30	3.75
Molybdenum carbide (Mo_2C)	4.50	3.45
Nickel	3.00	1.90
Tantalum carbide (TaC)	2.65	8.00
Tin	2.45	3.15
Titanium carbide (TiC)	3.20	3.65
Tungsten		
Fine	1.15	4.45
Coarse	6.00	6.10
Tungsten carbide (WC)		
Fine	1.45	4.20
Coarse	6.50	6.50
Tungsten-titanium carbide (WC-TiC)	3.90	4.30
Vanadium carbide (VC)	4.50	2.70
Zirconium	3.70	2.50

Permeability and Flow Rate

Sizing a hopper outlet or feed tube to achieve the required discharge rate is more difficult, but no less important, than overcoming arching, particularly for fine powders. All bulk materials have a maximum rate at which they discharge through a hopper opening of a given size (e.g., a Hall flowmeter test). For example, for free-flowing bulk materials, a good approximation of this maximum discharge rate of a coarse material (e.g., 3 mm and larger particles), from a mass flow hopper can be predicted:

$$Q = \gamma A\{Bg/[2\,(1+m)\tan\theta]\}^{1/2}$$

where Q is the maximum steady discharge rate, γ is the bulk density, A is the cross-sectional area of outlet, B is the outlet diameter or width, g is the acceleration due to gravity, m is 1 for circular opening and 0 for slotted opening, and θ is the flow channel angle (measured from vertical) in degrees. This equation can be modified to take particle size into account, but this modification is only important if the particle size is a significant fraction of the outlet size (Ref 8).

Obviously most metal powders cannot be considered "coarse" materials; therefore, the above equation rarely applies in P/M applications.

For fine powders, funnel-flow bins often exhibit high discharge rates, but controlling the flow rate is always a challenge because the flow channel is not likely to be stable. As a result, the actual size and shape of the stagnant region is neither well defined nor constant. The flow channel can change size radically or collapse, creating flow rates that range from no-flow conditions to complete flooding.

Fine powders are more easily handled in a mass flow bin, whose flow channel is stable and predictable. Because all of the material is constantly moving in a mass flow bin, the flow channel is set by the shape of the bin.

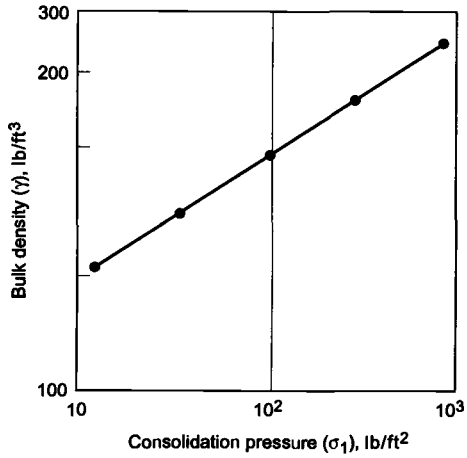


Fig. 18 Bulk density as a function of consolidation pressure (metal powder). Increasing consolidation pressure increases the bulk density.

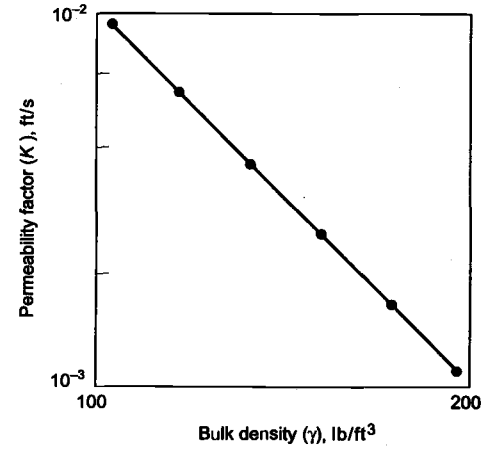


Fig. 19 Permeability as a function of bulk density (metal powder). Increases in bulk density reduce permeability of a material. Unless properly accounted for during bin selection, increased bulk density and reduced permeability can interrupt predictable flow.

However, it should be remembered that the maximum flow rate of a fine powder through the outlet of a mass flow bin is low compared with that of a coarse, granular solid. For fine materials, the expansion and contraction of voids during flow can create an upward air pressure gradient at the outlet of a mass flow bin. During discharge, this upward gradient acts against gravity, reducing the discharge rate. Such gradients do not usually form with coarser particle materials. Because coarse materials are more permeable than fine ones, air is allowed to flow freely into and out of the voids as they expand and contract.

This phenomenon can be analyzed by considering how gas flows through a bed of powder when a pressure differential occurs across the bed. When the gas velocity is low, flow through the bed is laminar, and Darcy's law can be used to relate gas velocities to gas pressure gradients within or across the bed. Darcy's law can be written in the following form:

$$u = -K\left[\frac{(dp/dx)}{\gamma}\right]$$

where K is the permeability factor of the bulk solid, u is the superficial relative gas velocity through the bed of solids, γ is the bulk density of the solid in the bed, and dp/dx is the gas pressure gradient acting at the point in the bed of solids where the velocity is being calculated. The permeability factor, K, has units of velocity and is inversely proportional to the viscosity of the gas. A permeability test is run by passing air (or other suitable gas) through a representative column of solids. The pressure across the bed is regulated, and the rate at which the gas flows is measured.

This approach allows the permeability of the bulk solid to be determined as a function of its bulk density. Figure 19 shows the test results for a sample of metal powder.

Because mass flow bins have stable flow patterns that mimic the shape of the bin, permeability values can be used to calculate critical, steady-state discharge rates from mass flow hoppers. Permeability values can also be used to calculate the time required for fine powders to settle in bins and silos and to design solids processing vessels to purge, heat, dry, or condition bulk solids.

Testing of Flow Rate

Flow of metal powders is determined by standard methods developed by ASTM and MPIF. Flow rate is the time required for a powder sample of a standard weight (50 g) to flow under atmospheric conditions through a funnel into the cavity of a container or mold. A determination of the flow rate of a powder is important in high-volume manufacturing, which depends on rapid, uniform, consistent filling of the die cavity. Poor flow characteristics cause slow and nonuniform press feeding and difficulty in ensuring even fills of the die cavity.

Before a powder is used in production, its flow characteristics must be known because some compacting tools require a free-flowing powder, while others can be used with a relatively poor-flowing powder. The term free-flowing refers to those physical properties of a powder—such as composition, particle fineness, and particle shape—that permit the powder to flow readily into the die cavity.

If a compacting tool is designed to handle a free-flowing powder, the use of a poor-flowing powder will necessitate modification. Compacting press manufacturers provide modified hopper designs and feeding shoe arrangements to accommodate finer, poor-flowing powders such as tungsten, molybdenum, or lighter aluminum powders.

The flow of powder from the feeding shoe into the die cavity can be increased by tapping or by changing the design of the filling device. These factors, however, are not taken into consideration in conventional flow rate test procedures, which consist of determining the time required for a given weight or volume of powder to flow through a standardized funnel-shaped cup with a small orifice at the bottom.

Table 6 Flow rate of metal powders through Hall and Carney funnels

Metal powder		Lubricant		Apparent density, g/cm³	Flow rate of 50 g (2 oz) powder, s		Flow rate of 25 cm³ (1.5 in.³) powder through Hall funnel, s	Weight of 25 cm³ (1.5 in.³) powder used in volumetric flow rate study		Calculated flow rate for 50 g (2 oz) powder based on volumetric flow rates, s
Material	Grade	Type	Additions, wt %		Hall funnel	Carney funnel		g	oz	
Iron	MP-35HD	Zinc stearate	None	2.81	25.77	4.62	37.74	70.27	2.46	26.86
		Zinc stearate	0.25	3.12	23.37	4.16	...	...	...	...
		Zinc stearate	0.50	3.05	25.93	4.30	...	...	...	...
		Zinc stearate	0.75	3.02	26.80	4.41	...	...	...	...
		Zinc stearate	1.00	3.00	27.57	4.59	40.55	74.52	2.61	27.21
Iron	MH-100	Zinc stearate	None	2.48	30.14	5.26	38.61	62.06	2.17	31.11
		Zinc stearate	0.25	2.97	23.23	4.14	...	...	...	...
		Zinc stearate	0.50	2.93	26.39	4.47	...	...	...	...
		Zinc stearate	0.75	2.86	28.97	4.80	...	...	...	...
		Zinc stearate	1.00	2.87	30.42	5.12	42.20	71.31	2.50	29.59
Iron	A-Met 1000	Zinc stearate	None	2.94	26.24	4.34	39.16	73.91	2.59	26.49
		Zinc stearate	0.25	3.27	23.89	4.04	...	...	...	...
		Zinc stearate	0.50	2.98	28.30	4.55	...	...	...	...
		Zinc stearate	0.75	3.18	25.46	4.41	...	...	...	...
		Zinc stearate	1.00	3.18	25.58	4.45	41.70	79.57	2.78	26.20
Stainless steel	304-L	Lithium stearate	None	2.61	30.62	4.92	39.65	65.45	2.29	30.29
		Lithium stearate	0.50	3.08	29.43	4.80	...	...	...	...
		Lithium stearate	0.75	3.01	33.20	5.42	...	...	...	...
		Lithium stearate	1.00	3.02	37.51	6.13	59.89	75.54	2.64	39.64
Premix bronze	5099	Stearic acid	None	2.96	21.68	3.99	32.69	74.92	2.62	21.82
(90% Cu–10% Sn)		Stearic acid	0.25	3.54	24.01	5.17	...	...	...	...
		Stearic acid	0.50	3.54	24.66	5.38	...	...	...	...
		Stearic acid	0.75	3.42	27.91	5.24	49.84	85.68	3.0	29.08
		Stearic acid	1.00	3.38	34.75	7.20	...	...	...	...
Brass	B-126	Lithium stearate	None	2.89	33.26	5.51	48.56	72.67	2.54	33.41
		Lithium stearate	0.25	3.06	33.52	5.77	...	...	...	...
		Lithium stearate	0.50	3.14	38.70	6.38	64.01	78.25	2.74	40.90
Aluminum	...	None	...	1.19	66.43	...	39.23	29.73	1.04	65.97

The following sections briefly review flow-rate test methods and variables that affect flow rate. Additional coverage is available in Ref 9 to 21.

Hall Flowmeter. The device most commonly used for measuring flow rate is the Hall flowmeter (Fig. 15a), taken from ASTM B 213 and MPIF 3 (equivalent standards include ISO 4490, Japanese standard JIS 7-2502-1966, and German standard 82-69). The test equipment consists of a funnel with a calibrated hole 2.5 mm (0.1 in.) in diameter. The funnel, which is made of aluminum alloy 6061-T6, is supplied with a smooth finish to minimize wall friction.

With the help of a stopwatch and weighing balance, the flow rate of metal powders can be easily determined. A dry 50 g weight sample is transferred to the funnel, the orifice of which is covered with the operator's fingertip. The stopwatch is started when the fingertip is removed and is stopped when the last quantity of the powder leaves the funnel. The flow rate (s/50 g) of the sample is reported as the elapsed time in seconds for 50 g of powder to flow through the orifice. A powder that does not flow through a 2.5 mm (0.1 in.) orifice Hall funnel, with or without an external impulse, is said to be a non-free-flowing powder (as per ASTM B 213 and MPIF 3 method).

The Hall funnel is calibrated using a standardized powder (150-mesh Turkish emery grit), a sample of which is supplied with the equipment. The desired hole size is obtained by precisely honing a drilled hole until a satisfactory flow rate of emery powder is obtained.

A change in surface finish and the radius of the orifice (at the junction of the wall), buildup of material on the sidewalls of the orifice, or enlargement of the hole size due to continuous use can alter the standardization of the funnel. Verification of the calibration should be performed periodically by using the standardized emery powder. Calibration of the Hall funnel with Ballotini solid glass spheres, having particle size ranges of 0.090 to 0.102 mm (0.0036 to 0.004 in.) and 0.065 to 0.090 mm (0.0026 to 0.0036 in.) diameters, has yielded flow rates of 35.6 and 33.4 s/50 g, respectively.

The feed of powder to the die is handled on a volume basis. Thus, differences in apparent densities of powders can lead to considerable variations in the weight of material filling a given volume. A test for volumetric flow-rate determination is under investigation by ASTM. Table 6 lists the flow rates of metal powders for both weight and volume basis. The volumetric flow-rate tests were carried out using the Hall flowmeter. The data in Table 6 indicate that the readings for volumetric flow rate lie within a narrower range, as compared to the Hall flow rate.

Many factors can affect the accuracy of the results obtained with the Hall flowmeter, such as the moist finger of an operator (use of gloves can eliminate this effect), vibration of the surface on which the flowmeter is placed, humidity and temperature, condition of the sample, uniformity of the powder mix, and alternate use of the device for unlubricated and lubricated powders. Residual lubricant film left by a lubricated powder on the flow-meter wall or orifice can affect the subsequent flow test results of an unlubricated powder.

Other problems associated with its use include:

- *Segregation of the mixture:* Because only a small sample (50 g) is used, slight changes in particle size distribution can result in widely varying and misleading values.
- *Limited flowability information:* If a powder is too cohesive to flow through the funnel, no information on the flowability of the powder can be determined. It has been estimated that up to 40 to 50% of all powder mixes used in the P/M industry will not flow through the Hall flowmeter.
- *Meaningless results:* Even if a powder does flow well, the value obtained (s/50 g) is not meaningful for many design problems. It cannot, for example, be extrapolated to predict limiting press speed, limiting flow rate through the feed hopper, or other rate-limiting phenomena.

The attempt to combine measurements of two material flow properties (minimum orifice size and flow rate) results in a method that does not measure either one very well (Ref 22).

Carney Funnel. Certain characteristics of some metal powders, such as particle shape and size distribution and lower specific gravity, may affect the powders to such an extent that they will not flow through the Hall funnel. In this case, the Carney funnel (Fig. 15b), which has the same dimensions as that of the Hall, except for a larger orifice diameter of 5 mm (0.2 in.), can be used to obtain a relative measure of the flowability of nonfree-flowing metal powders. The use of the Carney funnel, which is further described in ASTM B 417 and MPIF 28, is not a standardized test method in that there is no standard calibration procedure. However, it is used in industry to compare flow rates through a 5 mm

(0.2 in.) orifice for a variety of materials. Because there is no correlating factor to relate the data obtained using the Carney funnel with that of the Hall, the user must establish an empirical relationship between the two methods.

Other Testing Methods. A number of other devices or methods for measuring the flow rates of metal powders have been developed. Efforts have been made to design test methods for powders that do not flow through either the Hall or Carney funnel and to reflect the shop floor conditions of these powders.

One method currently under consideration by the International Standards Organization determines the filling characteristics of metal powder into cavities of increasing sizes. A powder-filled shoe is slid back and forth once on the surface of a 40 mm (1.6 in.) thick plate (placed on a paper) into which bores with diameters of 5, 7, 10, 15, 20, 25, and 30 mm (0.2, 0.3, 0.4, 0.6, 0.8, 1.0, and 1.2 in.) have been drilled.

The plate is then lifted, and the powder that has fallen onto the paper from each of the seven die cavities is weighed. When the mass of the powder is divided by the volume of the respective cavity, the filling density for each cavity is obtained. The test provides the critical diameter, that is, the dividing line between cavity diameters which will be filled at a constant apparent density and those through which the powder may not flow at all or which may result in incomplete cavity fill.

In 1956, a second test method for determining the flow rate of metal powders that do not flow through the Hall funnel was developed by Chrysler Corporation as an internal standard. The test equipment consists of a powder shoe of 102 cm^3 (6.2 $in.^3$) in volume and a pivoted lever attached to the shoe at one end and to a cam follower at the other. A four-lobed cam is connected to a gearbox and is driven by a motor. A circular opening of 12.7 mm (0.5 in.) diam (with a 3.0 mm (0.12 in.) wide bar at center) drilled on a 2.4 mm (0.1 in.) thick metal plate acts as a die opening.

To conduct the test, the powder-filled shoe is moved back and forth over the opening four times. This is referred to as a one-cycle operation. The quantity of powder passing through the opening is collected on a balance pan and weighed. The test equipment simulates the action of a production press, and flow is measured in terms of the quantity of powder that passes through the opening in one filling cycle of a shoe.

In one production example, the normal range for a bronze powder mix was 80 to 95 g (2.8 to 3.3 oz) per cycle, whereas the data of all the lots ranged from 30 to 125 g (1 to 4 oz) per cycle of operation. The test method was used to check the flow of a production mix under conditions comparable to those encountered in the production presses. The test indicated the flowability of a powder mix accurately.

The die-filling operation of a production press can be simulated by a third test method in which actual bearing die cavities are used. For the production of bearings with varying wall thicknesses and complicated shapes, the tool design should accommodate the variations in filling properties of the powder mixes. The following test procedure was found to be especially useful for complex powder mixes that may or may not flow through the Hall flowmeter.

The equipment consists of a powder shoe, which slides over a metal plate with a recessed cavity in which a series of cups (with or without core rods) can be fitted. The sizes of the resulting bearing-shaped cavities vary between 3.3 to 8.2 cm^3 (0.2 to 0.5 $in.^3$) in volume with wall thicknesses ranging from 1 to 3 mm (0.04 to 0.12 in.) and corresponding core rod diameters from 19 to 15 mm (0.8 to 0.6 in.) with a constant height of 45 mm (1.8 in.). A reference cavity of the same height is coreless, with a volume of 16 cm^3 (1.0 $in.^3$).

With the selected size of the bearing die in position, the powder shoe is mechanically traversed through the die (or cup) opening and back to the original position. The cup is then removed and weighed. The quantity of powder is divided by the total volume of the cup, and the fill density of the powder is calculated. The process is repeated for the remaining die sizes by changing the core rod inserts. The filling density of the reference cavity is always the highest and is designated 100 for a given sample. This value is decreased with a reduction in wall thickness.

Tests with iron, nickel, copper, tin, and Turkish emery grit indicate that the results are consistent. The powders tested either flowed into the cup or did not enter at all. By evaluating filling densities in different die sizes, the test provides meaningful information in designing tools for a production shop. This test method was used to study blending variables and yielded close agreement with the performance of the production presses. Unfortunately, the test is not simple and requires the use of precise equipment.

Variables Affecting Flow Rate

Flow characteristics are dependent on several variables, including interparticle friction, particle shape and size, type of material, environmental factors, and weight of the bulk.

Characteristics of powder surfaces, such as surface oxide films and lubricant films, also affect flow characteristics. The presence of oxide films on powder particle surfaces alters the friction between particles and increases flow rate. Powders with lower surface oxide contents flow more slowly than powders with higher oxide levels. Minute additions of lubricants may increase the flow rates of metal powder, but further additions will reduce flow rates. For practical purposes, the higher the lubricant level, the slower the Hall flow rate (see Table 6).

In general, reduced flow rates are encountered with powders that exhibit one or more of the following characteristics: low specific gravity, low apparent density, high friction coefficient of fine particles, high specific surface area, a complex blend of different materials, and high moisture content.

Interparticle Friction. The resistance to flow depends primarily on the regions in which one particle hampers the free movement of other particles, either by direct contact or indirectly. This is mainly determined by the coefficient of interparticle friction. Particles may be prevented from moving separately by temporary adherence or interlocking. In this manner, clusters are formed that may occupy considerable volume. The phenomenon of cluster formation depends on the movement and type of powder, the flow varying markedly with the size and structure of the particles. If the particles were all truly spherical, they would generally roll readily into a die cavity. This can rarely be achieved in commercial powders; differences in size and shape are unavoidable.

Particle Size and Shape. Subsieve powders, those with particle sizes less than 44 μm, generally have poor flow rates. For this and other reasons, very fine powders are not used for compacts that are pressed on automatic presses. The particles of most powders used for compacting have irregular equiaxed shape, with flow rates between those of spherical (high flow rates) and flaky (low flow rates) powders.

Type of Material. Flow is influenced by the type of material, whether it be copper, aluminum, or iron. The major influence is the theoretical density. Other characteristics, such as adhesive and cohesive surface properties and magnetic or electrostatic interactions, are also factors.

Environmental Factors. Powders exposed to air containing high relative humidity absorb moisture on particle surfaces, resulting in reduced flow rate. On the other hand, as moisture content increases, many materials tend to agglomerate, which increases permeability and, consequently, increases discharge and settling rates. Seasonal changes in temperature do not affect the flow rate of metal powders considerably. Very low temperatures, however, can cause condensation of moisture; very high temperatures may partially melt the lubricants in the powder mix. Such extreme temperature conditions may cause interruptions in the flow of material through conveying systems. Also, because the permeability factor, K, is inversely proportional to the viscosity of the air or other gas in the void spaces, heating causes the gas to become more viscous, which makes the bulk solid less permeable.

Weight of the Bulk. A metal powder with a lower specific gravity, such as aluminum, generally exhibits slower flow rates compared to high specific gravity powders such as iron. At the same time, the higher the apparent density of a given material, the faster the flow. The ratio of apparent density to specific gravity can be used to correlate the bulk properties of various metal powders.

Sliding at Impact Points

Two key factors in chute design are the chute angle and the smoothness of the chute surface at

the point of impact. Too shallow or too rough a surface in a chute impedes flow.

The required minimum chute slope can be determined by placing a ring-type device containing a sample of the powder on a representative sample of the chute wall surface and applying a predetermined vertical load to simulate impact. Once the weight has been removed, the plate is raised to determine the angle at which the particles begin to slide (Ref 23).

All of the factors affecting frictional properties can influence the optimal chute angle, except for time of storage at rest. Because it is uncommon for a chute to remain full without material moving through it, that parameter is generally not a design consideration.

Segregation Tendency

Providing a reliable, uniform mix or blend is critical in producing quality P/M parts. This requires not only proper mixing methods, but also proper handling techniques through compaction to ensure a uniform delivery of the powder to the die. As such, much greater emphasis is now being placed on appropriate storing, feeding, and transporting of powders.

The exact method of die fill depends on the specific compaction process being used (e.g., hydraulic, mechanical, rotary, isostatic, etc.) but generally a feed shoe is used. At the beginning of a compaction cycle, the feed shoe indexes over the die (lower punch) in the fill position and delivers a predetermined amount of powder (sometimes with the assistance of vibration). This is the final and most critical point where variations in the delivered mix directly impact part quality.

Much attention has been given to this specific step in trying to aid in powder filling such that a uniform fill is ensured (Ref 24). However if demixing has already occurred upstream in the process, then the compact will be nonuniform with resulting part-to-part variations. For example, lubricant variations will impact wall friction and subsequent compacting pressures. This, in turn, will cause some variation in compact strength, composition, and density.

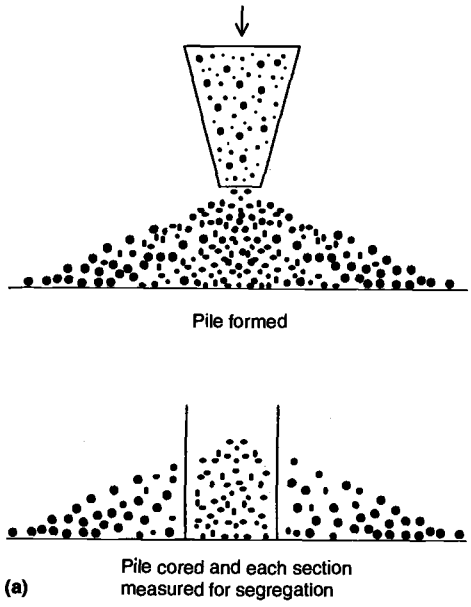
Even more critical may be the variation in particle size as it impacts packing behavior and introduces significant density variations. Considering all the other critical variables that affect the compact density and strength (Ref 25) (e.g., compaction pressure, compaction velocities, temperature, wall surface variations and wear, etc.), ensuring that a proper final mix is delivered into the die becomes critical to reducing process complexity and ensuring a high-performance process.

Mechanisms of Powder Segregation. Five mechanisms have been identified as the primary cause of most segregation problems in particulate materials (Ref 26). Of these five, four are known to occur with P/M powders, and each of these mechanisms is described below. The conditions that tend to promote each mechanism are described in Ref 27. Methods to test the tendency for two of the mechanisms to segregate a particular powder blend or mix are also described below. More complete descriptions may be found in Ref 7 and 28.

Sifting. This common phenomenon occurs as smaller particles move through a matrix of larger ones. One of the most likely places in typical P/M processes where sifting segregation can occur is in the filling of a container. For example, during the discharge of the blender (or container) into a portable container (or surge hopper), a concentration of the fine particles will develop under the fill point while the larger particles will tend to roll or slide to the periphery of the pile.

The tendency of a material to segregate by sifting can be determined by running a sifting segregation test. First, a pile is formed under controlled conditions. The pile is then cored to gather a representative sample of particles from its center (under the fill point) and periphery (Fig. 20a). Each cluster is evaluated for particle size distribution, chemical content, and other relevant variables in order to determine the degree of segregation that has occurred. Figure 20(b) shows the results of a sifting segregation test, using a binary mixture.

Particle Velocity on a Surface. In general, the frictional drag on particles moving on a chute surface is higher for finer particles than for coarser particles. This results in a velocity difference between the coarse and fine particles, which translates to differences in particle trajectories off the end of a chute. The higher drag of the finer particles along with their position near the chute surface results in a concentration of the finer particles nearer to the end of the chute



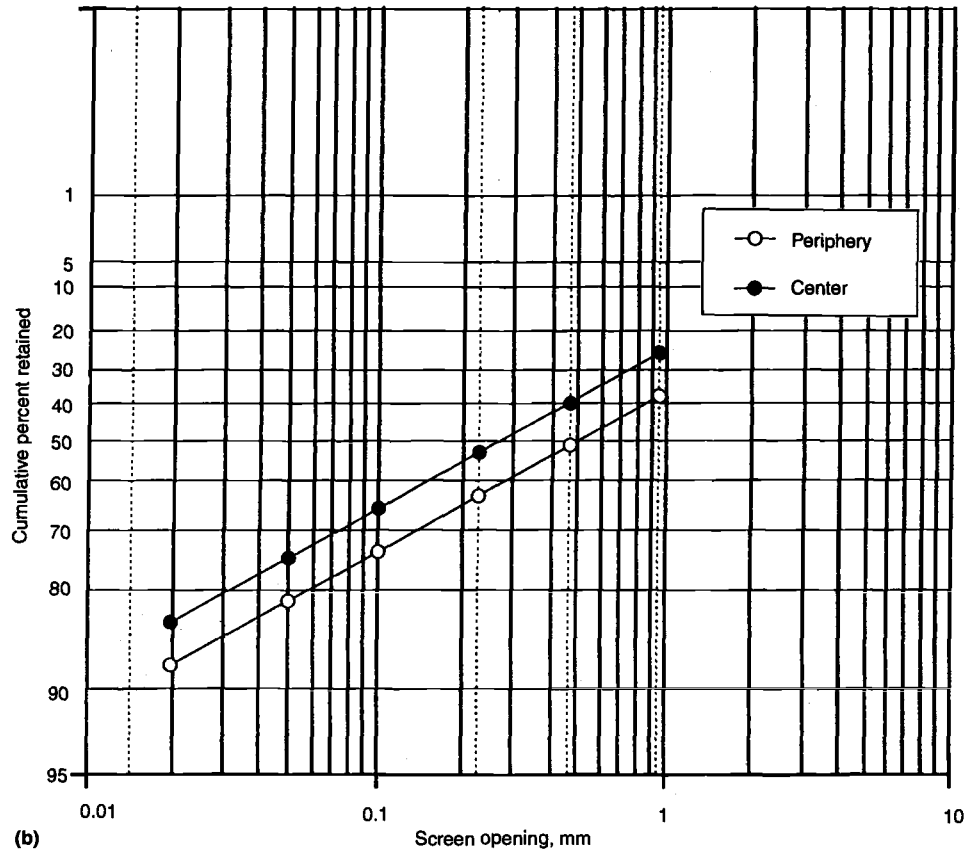


Fig. 20 Sifting segregation test. (a) Test setup. (b) Test results for binary mixtures

while the coarser particles come to rest at a much greater distance.

This can become particularly detrimental to the compacting process if portions of the powder stream go to multiple die cavity inlets or if the die cavity provides enough space for this trajectory segregation. In addition, if there are chutes within any of the powder transfer steps, there is the potential for this mechanism to occur.

Air Entrainment (Fluidization). In general, fine or light particles are less permeable than coarse or heavy ones. This allows the finer/lighter particles to retain air longer in their void spaces. Thus, when a mixture of coarse/heavy and fine/light particles is charged into a container or bin, down an empty vertical standpipe, or potentially into the die cavity, the finer particles remain fluidized longer while the coarser particles settle first (sometimes referred to as sedimentation). This results in a vertical segregation pattern within the powder bed.

To determine the likelihood of segregation by air entrainment, a tall cylinder containing a sample of the solid is aerated for a short period (Fig. 21). After the fluidizing air is turned off, sections of the cylinder are removed and the contents of each section are analyzed. If the material has segregated by air entrainment, fines will be located near the top of the cylinder and the coarser or heavier particles will be located at the bottom.

Particle Entrainment. Fine/light particles tend to remain suspended in air longer than coarse/heavy particles. Thus, for example, air currents can carry airborne fines and light particles away from a fill point to certain areas of a bin, such as toward vents and dust collectors.

Variables that can affect the tendency of particles to segregate include:

- *Particle size distribution and shape:* Sifting segregation is most likely to occur when the material has a range of particle sizes and when there is interparticle motion during operation. Generally, particles greater than about 100 μm in diameter are most susceptible to sifting segregation. If most of the particles are smaller than 100 μm, segregation is more likely to occur by air or particle entrainment.
- *Cohesiveness:* The more cohesive a material, the less likely it is to segregate. Thus, for some materials, the potential for segregation can be reduced by adding binders. In general, this makes the material less free-flowing and causes fine particles to stick to coarse ones. Caution is advised, however, because the additive can rapidly change a situation from one of free flow to one of no flow.
- *Bin flow pattern:* The type of flow pattern that develops can significantly affect the segregation tendency of materials. Typically, funnel flow patterns exacerbate side-to-side segregation (such as that caused by sifting), whereas a mass flow pattern tends to minimize such problems.

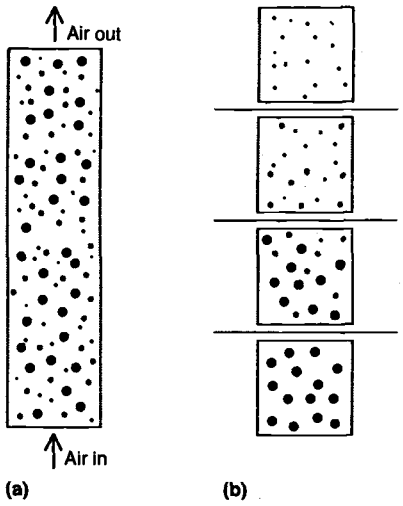


Fig. 21 Fluidization segregation test. (a) Column of material fluidized. (b) Column split and each section measured for segregation

Angle of Repose

The angle of repose of an aggregate, or the angle of the surface of an unconstrained pile of solids with the horizontal, is of practical interest rather than of theoretical concern. Of the various physical properties of bulk powders, the angle of repose is the easiest to obtain. It is related to interparticle friction and the flowability of cohesion-less material. This property is frequently used to characterize powdered materials.

The angle of repose of a powdered material does not always give satisfactory reproducibility and is often masked by other factors that are not inherent to the material (most frequently, the presence of a liquid). Thus, before a standard measuring method that provides reasonable reproducibility is developed, its usability as a measure of powdered material property must be established.

However, although the angle of repose is frequently used as a convenient characterization of powdered material, its ultimate reliability depends on the measuring method used. Therefore, the measuring method should be carefully selected, especially when the measured angle of repose is to be used to determine another property, so that it best reflects the property to be quantified.

Measurement Methods

Several methods have been used to measure the angle of repose of materials. Train (Ref 29) studied the angle of repose of a number of grades of glass spheres, lead shot, and silver sand using four different methods, as illustrated in Fig. 22. These methods are:

- *Fixed height cone:* The powder is carefully poured through a funnel at a fixed height until the apex of the heap formed by the powder reaches the tip of the funnel. The tangent of the angle of repose is the ratio of the height to the mean radius of the base of the powder heap.
- *Fixed base cone:* The powder is allowed to flow through a funnel, which is raised vertically until the heap covers a circular base of fixed size. The tangent of the angle of repose is calculated in the same manner as the fixed height cone method.
- *Tilting table:* A rectangular box filled with powder is tilted until the contents begin to slide. The angle that the upper surface of the box makes with the horizontal is equal to the angle of repose.
- *Rotating cylinder:* A sealed hollow cylinder half full of powder is rotated until the surface of powder exhibits its maximum angle with the horizontal. This maximum angle is the angle of repose.

Brown (Ref 31) reported three methods to measure the drained angle of repose (Fig. 23) and a rotating drum method to measure the dynamic angle of repose, as follows:

- *Ledge:* Material is first charged into a rectangular Perspex box that is 30 cm (12 in.) in

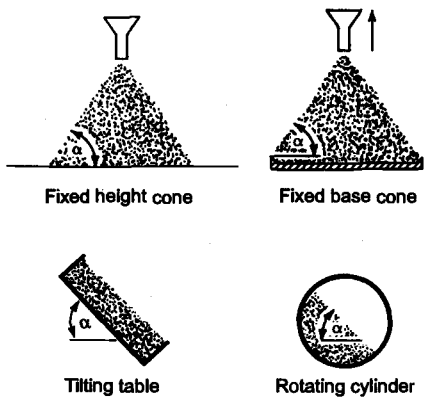


Fig. 22 Four methods used to measure the angle of repose. Source: Ref 30

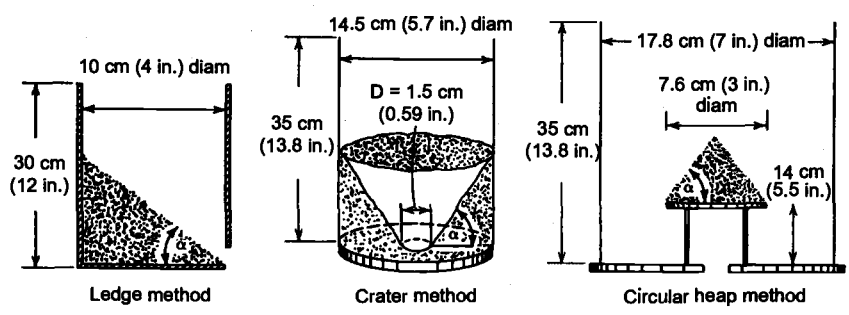


Fig. 23 Apparatus used to measure the drained angle of repose. Source: Ref 30

Table 7 Drained angles of repose (degrees to the horizontal)

Descriptive class	Material	Circular heap (±1½°), α	On a ledge (±1½°), α	Crater (±2°), α	Dynamic(a), α
Smooth, spherical	Beads	17½	25	27	25½
	Beads	20	23	21½	24
Rough, nearly spherical	Sand	32½(b)	34	35–35½	34½
	Tapioca	30	34	37½	32
	Rice	35	37	42	...
Angular	Sand	37	37	39	36½
	Sand	35½	37	38½	38½
	Sand	35½	36	37½	38½
	Durite	37	40	41	...
	Charcoal	...	38	...	...
	Charcoal	...	38½	...	...
	Charcoal	...	42½	...	...
	Charcoal	...	42½	...	...
	Coal	37½	37	41	34
	Coal	35½	37½	...	...
	Coal	36	38	...	...
	Coal	36½	38½	...	...
	Coal	38½	38½	...	...
Containing fine particles	Coal	...	52	...	...
	Coal	54	59–61	...	...
	Coal	...	47½	...	...
	Fine coal	...	67	...	...
	Limestone	...	64	...	...

(a) In a drum rotating at 6 rpm. (b) On a 5.1 cm (2 in.) diam platform in a 12.7 cm (5 in.) diam cylinder. Source: Ref 30

height with a 10 by 10 cm (4 by 4 in.) base. A slot at the base of one vertical wall can be closed by a board. The closure board is then removed to allow the material to flow slowly through the narrow slot. The angle with the horizontal of the surface of the material remaining when the flow stops is measured as the angle of repose.

- *Crater:* A circular Perspex tube with a 14.5 cm (5.7 in.) diam is place vertically on a flat, horizontal base plate having a 1.5 cm (0.59 in.) diam orifice in the center. The powder is discharged through the orifice. The height of the remaining material resting against the wall of the tube is measured at eight equidistant points around the circumference to determine the angle of repose.
- *Circular heap:* A circular platform 7.6 cm (3 in.) in diameter is supported horizontally over a circular hole in a flat base plate and surrounded by a cylindrical tube having 17.8 cm (7 in.) diam and sufficient height (35 cm, or 13.8 in.) to ensure that when it is filled with powder the platform and any heap that may form on it is completely immersed in the powder. The powder in the cylindrical tube is then allowed to flow slowly out of the circular hole in the base plate. The height of the resulting heap on the circular platform is then obtained. The tangent of the angle of repose is calculated as in the fixed height methods.
- *Dynamic angle of repose:* A drum 15 or 30 cm (6 or 12 in.) in diameter and 10.2 cm (4 in.) long with Perspex end faces and roughened internal surfaces is half filled with powder and slowly rotated counterclockwise, with its axis horizontal. Within a certain range of rotation speeds (usually 2.5 to 6 rpm), the surface of the powder in the drum becomes substantially steady. The angle of inclination of the surface to the horizontal is measured at various speeds to determine the angle of repose.

Table 7 lists the angles of repose measured using these methods. Henein et al. (Ref 32, 33) used a method similar to that used by Brown to determine the dynamic angle of repose and lower angle of repose (shear angle) of several materials. Rotating cylinders of 40 and 106 cm (16 and 42 in.) diam were lined with 24-3 grit type E silicon carbide abrasive paper. One of the two end plates of each cylinder were made of plexiglas to observe and photograph the bed. The maximum angle of bed inclination just before slump occurred was measured with a long-arm protractor, and this angle was designated the upper angle of repose, or the dynamic angle of repose. The angle relative to the horizontal of the shear plane that separated the slumping solids of the bed surface from the material moving with the cylinder wall was considered the lower angle of repose, or shear angle (see Fig. 24). Table 8 summarizes the upper and lower angles of repose of several materials.

Riley et al. (Ref 34) reported methods to measure the angles of repose of cohesive powders. They used both tilting box and fixed bed methods to measure the angle of repose of mixtures of glass ballotini and cohesive powders of different compositions and then extrapolated the results to obtain the angles of repose of pure cohesive material. Figure 25 shows a sample plot and gives the angle of repose of several cohesive powders.

Factors Affecting Angle of Repose

Internal factors are those inherent to powders, or characteristic of the nature of powders. These include particle size, particle shape, and cohesiveness. In general, larger particles have higher angles of repose. However, very small particles may exhibit cohesiveness due to the electrostatic effect, which increases the angle of repose (Ref 31–33). Because spherical particles have a greater tendency to roll, they typically have smaller angles of repose than irregularly shaped particles.

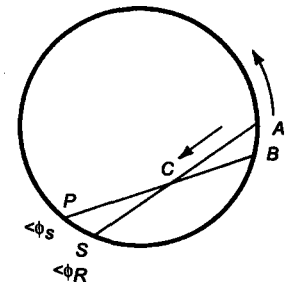
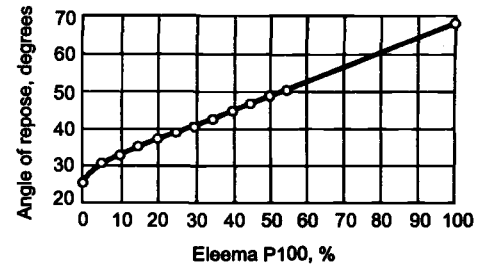


Fig. 24 Dynamic angle of repose and shear angle of material in a rotating cylinder. *B*, shear plane; ϕ_R, upper angle of repose (dynamic angle of repose). Angle ϕ_S, is lower angle of repose.



Powder	Tilting box	Fixed bed
Calcium carbonate	83.0	78.0
Eleema P100 (microfine cellulose)	64.5	68.0
Avieel PH101 (microcrystalline cellulose)	57.5	51.5
Methylcellulose 20 BPC	50.0	52.0
Methylcellulose 450 BP	63.0	55.0
Hydroxypropyl methylcellulose 4500	64.5	...
Hydroxypropyl methylcellulose 5000	59.5	58.5
Hydroxyethyl methylcellulose 3500	61.0	61.0
Magnesium stearate	69.0	66.0
Stearic acid	...	63.5

Source: Ref 34

Fig. 25 Typical plot of mixture composition versus angle of repose. Curve extrapolated to determine angle of pure material. Source: Ref 34

External factors are introduced by the environment and may include the method of measurement or the presence of other materials (either solid or liquid). The effect of measuring method is clearly evident as discussed above. In general, the more momentum introduced by the measuring method, the smaller the angle of repose. As a consequence, the drained angle of repose is higher than that obtained from the heap formation method.

The angle of repose of loosely packed dry powder may be increased by compacting, as well as by introducing liquid to the powder. Figure 26 demonstrates the significant effect these factors have on the angle of repose (Ref 30, 35).

Frydman (Ref 36) reported that the angle of repose of potash pellets may be increased by

Table 8 Upper and lower angles of repose of several materials

Material	Average size mm	Average size in.	Particle shape	Particle density, kg/m³	Loose bulk density, kg/m³	Dense bulk density, kg/m³	Static angle of repose, degrees	Dynamic angle of repose, degrees	Cylinder diameter m	Cylinder diameter ft	Shear angle, degrees
Gravel	3.0	0.12	Angular	2870	1560	1690	40.7	37.5	0.40	1.3	34.7
								37.0	1.06	3.5	34.4
Iron oxide	11.6	0.46	Spherical	...	...	...	31.5	35.2	0.40	1.3	33.3
Limestone B	4.3	0.17	Irregular	2700	1450	1610	40.3	39.6	0.40	1.3	37.7
								36.5	1.06	3.5	34.5
Limestone C	1.5	0.06	Irregular	2690	1520	1600	37.8	36.0	0.40	1.3	33.6
									1.06	3.5	32.5
Limestone D	0.58	0.02	Irregular	2680	1490	1570	35.6	34.9	0.40	1.3	33.5
Limestone F	8.1	0.32	Angular	2690	...	...	42.8	41.5	1.06	3.5	38.5
Nickel oxide	4.9	0.19	Spherical	...	870	900	32.5	30.2	0.40	1.3	29.9
Sand B	0.50	0.02	Nodular	2660	1640	1740	33.4	33.6	0.40	1.3	32.2

Note: 1000 kg/m³ = 1 g/cm³. Source: Ref 33

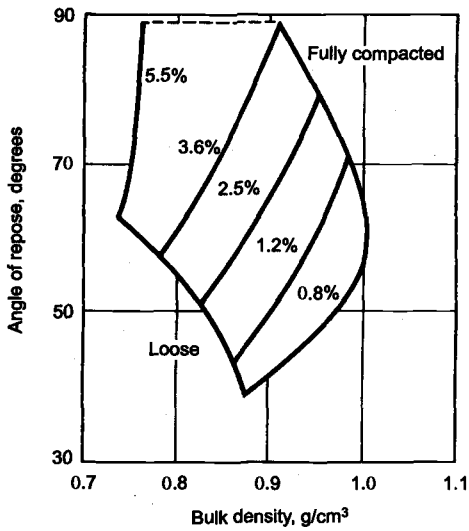


Fig. 26 Effect of moisture content on angle of repose of loose and compacted coal. Source: Ref 30

more than 100% by applying a light spray of moisture to the material surface. The effect of the presence of another solid is shown in Fig. 25.

REFERENCES

1. J.H. Bytnar, J.O.G. Parent, H. Henein, and J. Iyengar, Macro-Segregation Diagram for Dry Blending Particulate Metal-Matrix Composites, *Int. J. Powder Metall.*, Vol 31 (No. 1), 1995
2. *Properties and Selection: Irons and Steels,* Vol 1, *Metals Handbook,* American Society for Metals, 1978
3. A.W. Jenike, Storage and Flow of Solids, *Eng. Exp. Station Bull.*, No. 123, University of Utah, 1964
4. J. Marinelli and J.W. Carson, Solve Solids Flow Problems in Bins, Hoppers and Feeders, *Chem. Eng. Progr.*, May 1992, p 22–28
5. "Standard Shear Testing Method for Bulk Solids Using the Jenike Shear Cell," D 6128, ASTM, 1997.
6. A.R. Poster, Ed., *Handbook of Metal Powders,* Reinhold, 1966, p 17
7. J.W. Carson and J. Marinelli, Characterize Bulk Solids to Ensure Smooth Flow, *Chem. Eng.*, Vol 101 (No. 4), 1994
8. W.A. Beverloo, H.A. Leniger, and J. Van de Velde, The Flow of Granular Solids through Orifices, *Chem. Eng. Sci.*, Vol 15, 1961, p 260–269
9. J. Haertlein and J.F. Sachse, The Flow Rate of Metal Powders, *Handbook of Metal Powders,* Reinhold, 1966
10. A. Adler, Flow Properties of Metal Powders, *Int. J. Powder Metall.*, Vol 5 (No. 1), 1969, p 7–20
11. G. Matei, N. Claussen, and H.H. Hausner, Influence of Relative Humidity on Flow of Metal and Ceramic Powders, *Modern Developments in Powder Metallurgy,* Vol 8, Metal Powder Industries Federation, 1974, p 5–11
12. A.V. Zborovski, Correlation between the Apparent Density of Iron Powder and Its Flow Rate and Pressing Behavior, *Poroshk. Metall.*, Vol 84 (No. 12), Dec 1969, p 5–9
13. R.O. Grey and J. K. Beddow, On the Hausner Ratio and Its Relationship to Some Properties of Metal Powders, *Powder Technol.*, Vol 2, 1968–1969, p 323–326
14. M.J. Mahoney, "Method for Determination of Flow Rates of Metal Powders Blends," Laboratory Test 301–302, Amplex Div., Chrysler Corp., 1956
15. "Determination of Filling Characteristics," TC-119/SC-2N 234, International Standards Organization, 1981
16. P.E. Matthews, "Volumetric Flow Rate—Experimental," B 09:02, ASTM, 1980
17. J. Oakley, A Method of Assessing the Die-Filling Characteristics of Powders, *J. Inst. Met.*, Vol 87, 1958–1959, p 26–28
18. H.H. Hausner, Environmental Effects on the Behavior of Metal Powders, *Int. J. Powder Metall. Powder Technol.*, Vol 14 (No. 4), 1978
19. D. Yarnton, Effect of Lubricant on the Flow and Packing Density of Cu Powders, *Eng. Mater. Des.*, Sept 1970, p 1103–1107
20. "Flow Rate of Metal Powders," B 213, ASTM, 1977
21. "Determination of Flow Rate of Metal Powders Using the Hall Apparatus," No. 03, Metal Powder Industries Federation
22. J.W. Carson, Applying Bulk Solids Flow Principles to Metal Powders, *Int. J. Powder Metall.*, Vol 11 (No. 4), 1975
23. D. Stuart-Dick and T.A. Royal, Design Principles for Chutes to Handle Bulk Solids, *Bulk Solids Handl.*, Sept 1992, p 447–450
24. L.R. Lawrence and J.K. Beddow, Powder Segregation During Die Filling, *Powder Technol.*, Vol 2, 1968–1969
25. R.M. German, *Powder Metallurgy Science,* Metal Powder Industries Federation, 1984
26. J.W. Carson, T.A. Royal, and D.J. Goodwill, Understanding and Eliminating Particle Segregation Problems, Vol 6 (No. 1), *Bulk Solids Handl.*, 1986
27. B.H. Pittenger, H. Purutyan, and T.A. Royal, Eliminating Segregation Problems in Powdered Metal Processing, Reprinted from *Advances in Powder Metallurgy & Particulate Materials—1995,* Vol 1, Metal Powder Industries Federation, p 111–121
28. J.C. Williams, The Segregation of Particulate Materials, A Review, *Powder Technol.*, Vol 15, 1976
29. D. Train, Some Aspects of the Property of Angle of Repose of Powders, *J. Pharm. Pharmac.*, Vol 10 (Suppl), 127T–135T, Dec 1958
30. R.L. Brown and J.C. Richards, *Principles of Powder Mechanics,* Pergamon Press, 1970
31. R.L. Brown, Flow Properties, Powders in Industry, *Soc. Chem. Ind.*, Monograph No. 14, 1960, p 150–166
32. H. Henein, J.K. Brimacombe, and A.P. Watkinson, Experimental Study of Transverse Bed Motion in Rotary Kilns, *Metall. Trans. B,* Vol 14, 1983, p 191–205
33. H. Henein, J.K. Brimacombe, and A.P. Watkinson, The Modeling of Transverse Solids Motion in Rotary Kilns, *Metall. Trans. B,* Vol 14, 1983, p 207–220
34. G.S. Riley, S. Mann, and R.O. Jesse, Angles of Repose of Cohesive Powders, *J. Powder Bulk Solids Technol.*, Vol 2, 1978, p 15–18
35. E.F. Wolf and H.L. Hohenleiten, Experimental Study of the Flow of Coal in Chutes, *Trans. Am. Soc. Mech. Eng.*, Vol 67, 1945, p 585–599
36. S. Frydman, The Angle of Repose of Potash Pellets, *Powder Technol.*, Vol 10, 1974, p 9–12